

2C-H iodination by way of silver salt facilitated *in situ* formation of methanesulfonylhypoiodite. A route to 2C-I or DOI

Preface

2C-H sulfate was prepared as in the write-up for 2C-B.

Eventually I decided to use 30 g of the 2C-H to make 2C-I. Some was left to make 2C-C in the next instalment.

For this synthesis, a bit more planning is needed than with 2C-B, where the bromination agent, NBS, is commonly found. The ingredients for this experiment are more expensive and harder to come by. Some of the reagents could fall into the “exotic” category. But otherwise it is all straightforward.

Let's take a look at the reagents first:

Chem Info

2C-H: 2-(2,5-Dimethoxyphenyl)ethan-1-amine or 2,5-Dimethoxyphenethylamine, $C_{10}H_{15}NO_2$

Mol. weight: **181.232 g/mol** (freebase), **217.69 g/mol** (HCl salt), **230.27 g/mol** (hemisulfate or sulfate salt). The sulfate salt is not very soluble in solvents like acetonitrile or DCM, but is soluble in water, alcohols. It remains to be seen whether 2C-H HCl is soluble in DCM, it is soluble in ethylene glycol and triethylene glycol.

pK_A is about 9.6, melting point of the HCl is 138 – 139 °C (Shulgin)

Silver: Ag

Yes, the stuff in 2nd place Olympic medals, you too can almost be a champion!

Mol weight: 107.87 g/mol. Hard metal, high melting point. If you decide to use elemental silver and make silver mesylate, get the powder form.

Iodine: The heaviest of the stable halogens, found in the metallic form as the diatomic I_2 with a

Mol weight: **253.81 g/mol** (diatom), Violet-black crystalline solid with a metallic luster, melts at 113.7 °C.

Soluble in DCM, chloroform, methanol, ethanol, diethyl ether, acetonitrile, and benzene. Not very soluble in water, isopropanol.

Silver methanesulfonate: silver mesylate, CH_3AgO_3S

Mol weight: **202.96 g/mol** CAS: 2386-52-9

Light sensitive and air sensitive, expensive. Better to make your own.

It was found that you can skip this and use a more commonly found silver salt. Use silver nitrate or even silver carbonate. The advantage of silver methanesulfonate is that it is more soluble than other silver salts, and seems to give a good yield.

A white and shiny fluffy powder. Dissolves well in water, dissolves ok in DCM.

Hydrogen peroxide: H_2O_2

Found as a solution in water, has that nice peroxide, hospital smell.

34.014 g/ mol of the pure stuff that is unstable.

30% solution contains 9.8 mol/ L, 35% contains 11.68 mol/ L. Be careful handling the concentrated solutions, with heating it decomposes quicker and quicker into water and oxygen. But safe when under 50 °C. Will use to form silver oxide, inexpensive, but could be unavailable at concentrations over 12%, need the 30% or 35% solution.

Methanesulfonic acid: (MsOH or MSA) $\text{CH}_3\text{SO}_3\text{H}$

Used to form silver mesylate from the silver oxide in situ. Miscible with water, methanol. A clear, colourless liquid with a pK_A of -1.9.

Mol. weight: 96.10 g/ mol. Use the 70% aq. solution commonly found, with **10.8 mol/ L**. Heavy, with a density of 1.35 g/ cm³ (the 70% solution). Cheap stuff as well.

Acetic acid (glacial acetic acid, GAA): Mol. weight: **60.052 g/mol**, density of liquid form: 1.049 g/ cm³, colorless liquid.

Potassium iodide: KI

Optional, to test for the presence of hydrogen peroxide when making the silver methanesulfonate reagent, can be easily made from KOH and Iodine.

Hydrochloric acid (HCl), to make the final salt form of 2C-I. Get the fuming 35 – 38% variety to dilute, with a molarity of **11.64 mol/ L**

Another solvent in the procedure is methanol (**MeOH**) to dissolve the iodine, which itself is miscible with **DCM** (dichloromethane) used to dissolve the silver salt. Quality **IPA** (isopropanol) and ethyl acetate (**EtOAc**) to wash some of the intermediate and final products.

Miscellaneous

If you are making **DOI**, then the procedure will be the same, just begin with

2,5 – DMA (2,5-Dimethoxyamphetamine) instead of 2C-H and adjust for the mol. weight.

Mol weight of **2,5-DMA: 195.26 g/ mol (freebase)**, **231.72 g/ mol (HCl salt)**. There is no reason why this procedure shouldn't work on 2,5 – DMA to form DOI, and I will give iodination a go on it next time to confirm.

The plan was to follow the procedure in this paper:

<https://pubs.acs.org/doi/10.1021/acs.orglett.1c01530> and use acetonitrile as the solvent for the reaction. However, I made a total mistake assuming that 2C-H sulfate would dissolve in MeCN. It does not, the freebase most certainly will, but you can not just add NaOH to MeCN to freebase the 2C-H, turns out to be a big no no. Adding aqueous NaOH to MeCN will cause a hydrolysis reaction of the acetonitrile. So if you plan to work with MeCN, you need to pick up already freebase 2C-H with it – not

so easy as 2C-H in the presence of air will quickly turn into a carbonate. But maybe 2C-H HCl dissolves more readily...

The 2C-H was filtered and the acetonitrile was scrapped, I found it sketchy to work with, and foresaw possible difficulties in the work-up as well. For some reason switched to DCM. Freebased the 2C-H sulfate in water first and then picked it up with DCM.

TLC to follow the reaction was not fruitful, but a 9:1:0.1 mixture of DCM: MeOH: triethylamine is usable to spot 2C-H and 2C-I in the centre of the plate. If you wish to experiment, mix up 18 mL DCM, 2 mL methanol, and 0.2 mL triethylamine (TEA) and store in a reagent bottle to use as the TLC eluent. I was lazy but could roughly see that the 2C-H and (putative) 2C-I spots were on top of one another, it was too difficult to discern however. So really I was going blind in the reaction, could not tell when it was over. More on that later.

Silver costs a bit more than other reagents, but how much do you need?

From **30 g of silver powder** I managed to make 53.87 g silver methanesulfonate with a 95% yield. This is 0.265 mol, to react the 2C-H I used a 1.2 mol equivalent, which was too much, think 1.05 ml would do it, the silver doesn't really go anywhere, in the reaction it mixes well, and doesn't evaporate or anything.

So beginning with 30g silver, one should be able to react ~0.25 mol 2C-H (or other substrates waiting for their turn at iodination). If weighing for the HCl salt of 2C-H, this makes for **54.4 g**.

30 g Silver makes about 54 g silver sulfonate which you can use on about an equivalent amount of 2C-H in grams. The cost of 30 g silver powder is about 140 – 160€. But you can filter the silver after the reaction and I believe convert to black silver oxide.

Do this by dissolving in water, and add NaOH. Dry and then just mix this with MSA to form fresh silver methanesulfonate again. Haven't tried yet, but seems straightforward enough.

So silver, (or silver salts) does present an initial investment, but they are easily collected. Then cleaned and converted back. The precipitation quickly after the reaction begins could be mostly silver iodide. Maybe it is a good long term investment and the price of silver rises, one day you might even blow silver powder into your grandchildren's eyes.

Look at what silver species you can get. Is it silver or a silver salt such as silver nitrate, silver carbonate, maybe even silver sulfate. The reaction should proceed with any of these. Iodine should form bonds with the salts, but the salts will have different solubility profiles and possible rates of reaction.

Preparation of Reagents

– Preparation of KI –

Although mostly superfluous, and readily available to buy from chemical suppliers and chemists alike, I thought it would be interesting to prepare my own potassium iodide.

The balanced formula is:



For 30 g iodine (0.118 mol) you need 0.236 mol KOH or 13.26 g.

1) Dissolve the KOH in the minimum amount of water that the formed product (KI) can dissolve in. Why? Because the reaction also forms potassium iodate. KIO_3 is much less soluble in water than KI, especially when the water is cool. Initially I dissolved the KOH in 200 mL water but this was too much. Looking at the reaction 5KI are formed or $5/6^{\text{th}}$ of the molar amount of KOH or 0.196 mol or 32g, and $1/6^{\text{th}}$ KIO_3 is made or 0.393 mol or 8.42 g.

The solubility of KI is 127 g for 100 mL water at 0 °C rising to 144.5 g/ 100 mL at 20 °C

The solubility of KIO_3 is 4.6 g/ 100 mL water at 0 °C rising to 8.1 g/ 100 mL at 20 °C

We will heat the reaction so both will be a bit more soluble. So to make sure the formed KI is dissolved, we will need 22.15 mL water to keep the KI dissolved at room temp. With this amount just over 1 g of the by-product KIO_3 will remain dissolved and ~7.4 g should be filtered off.

In fact the procedure outlined here:

https://pdf.benchchem.com/1/An_In_depth_Technical_Guide_to_the_Synthesis_of_Potassium_Iodide_from_Potassium_Hydroxide_and_Iodine.pdf

Suggests to dissolve in twice the volume of water, 13.26 g dissolved in 26.5 mL water. So about the same.

2) Once the KOH is dissolved, heat with stirring, and begin adding the iodine, if the correct amount is used the solution should go close to clear very quickly, if it doesn't clear up add a bit more KOH. There will be colour left, but that is not from the iodine but likely from the KIO_3 . Slightly yellow water. The iodine binds to potassium and KI is white.

3) If you add too much KOH, add a bit more iodine, just watch how it clears up like a big boy.

4) Remove from heating and stirring, remove the magnet, and set to cool.

5) Filter over a Büchner funnel, and then fully fan dry the product. Should be white and sparkly crystals. So you may ask yourself why have I been preparing KI for 2 days? To protect yourself and your family from radiation of course. Congratulations, you now have 29.345 g of KI to play with (made from a bit over 30 g iodine and a bit over 13g KOH), albeit with just a bit of KIO₃ impurity present. A bit of impurity won't bother us when the end is near.

– Preparation of Silver Methanesulfonate –

The procedure to make this reagent was lifted from here:

<https://patentimages.storage.googleapis.com/5d/4e/3a/4d82f34a395cfa/EP0711753A1.pdf>

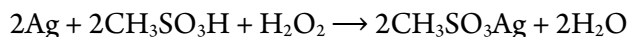
M. D. Gernon, *US Pat.* 5491247 or M. D. Gernon, *Eur. Pat.* EP 0711753A1

More information here:

<https://pubs.rsc.org/en/content/articlelanding/1999/gc/a900157c/unauth>

<https://annas-archive.gl/scidb/10.1039/a900157c/>

The balanced formula is:



In short, suspend a bit more than an equimolar amount of Ag in MSA, and then add hydrogen peroxide slowly watching for excessive foaming, then heat to 45 °C, once foaming settles down, add a second portion of peroxide and stir with heating increasing up to 50 - 60 °C until the layer is clear, free of silver. Protect the reaction from light throughout. Light will degrade the silver methanesulfonate as it forms.

An excess of peroxide is needed, 3 to 4x as most bubbles off as oxygen and does not react. In the “Environmental benefits of methanesulfonic acid” paper a 2x excess is added twice. The first addition is equimolar to the amount of silver which is a 2x excess in terms of the chemical formula and the reaction.

Procedure in detail:

1) **30.09 g of silver powder (0.279 mol)** is mixed with **60 mL 70% MSA (0.577 mol)**, note I used too much MSA here, thought it would not be enough to mix. Use less. Mix with a magnet in a 500 mL **amber glass reagent bottle** set in an ice bath. The ice bath is unnecessary as well, a cool room temp. is fine for the amounts described.

2) Drip in **47 mL 35% H₂O₂ (0.547 mol)** from an addition funnel. When the temperature is cold the peroxide is almost unreactive, but with every 10 °C increase in temperature H₂O₂ will react 2.2 x as fast, it will start to decompose more, and bubble more. Drip in slower if without an ice bath and watch for too much temperature increase. As well, starts to react with a delay.

3) Remove from ice bath and **increase the temperature to 35 °C**, watch for excessive foaming overwhelming your reaction vessel. But generally 30 g or 50 g is not much to react, the amount of MSA is small as well, the only real danger here is to not get the peroxide on your skin.

4) Ever so often take a sample with a micropipette and drip into a **10% KI solution**. Aha! So there was a use for the iodide, nuclear winter aside. Any excess peroxide will at once oxidize the colorless I⁻ iodide ion into iodine I₂, discolouring the solution. It is very sensitive. But I found that there was always some peroxide left unless maybe you vigorously boiled the mixture. Even after 2 days the iodide peroxide test came back positive.

5) Add the **second portion of H₂O₂** as before, could add a bit less, I dripped in 40 mL. Keep away from light, best to carry out the reaction in a dark room with foil wrapped on your device.

6) Heat to 50 °C and it slowly bubbles away. According to Google AI:
“80 mL of 35% (w/w) hydrogen peroxide releases approximately
10.42 liters of oxygen gas at Standard Temperature and Pressure (STP).”

So that is a lot to bubble off, maybe if none were consumed to form Ag₂O, and it bubbled off at once, yes then it could very well be violent.

But at 50 °C for 36 hours + was too long, the reaction layer was clear after a much shorter amount of time. One could heat more, to 60 or 80 °C, to speed things up but stay vigilant, protect ya neck, and I ain't got yo back.

7) Decide to pour on a plate and bubble off at 130 °C with a fan blowing. Still it goes very slowly, in fact water probably boils off quickly and what is left is a more viscous mixture. The scent of peroxide can be strong if there is a fan blowing on the plate.

8) Decide to risk filtering on a Büchner funnel. Finally, completion and progress, with no explosions. In the funnel, wash with a bit of IPA as well. End up with a white sparkly powder. Looks good. Weight is **53.868 g (0.265 mol), or a 95% yield**. A complete success... in making a reagent for the actual synthesis of the intended product...

9) Store the silver salt in a clean amber glass bottle to protect from light.

The Actual Synthesis You Have Been Waiting For

As written many pages back, the acetonitrile was jettisoned and I opt to use DCM instead after filtering and drying the 2C-H sulfate. And proceed with a synthesis possibly yet untested by mankind.

- 1) Dissolve about **30 g 2C-H sulfate (0.13 mol)** in 300 mL water, but this was far too much, could use even maybe 100 mL and freebase with a stoichiometric amount of NaOH or **5.2 g** dissolved in about 80 mL water. Believe the water went opaque as freebase oil became trapped in the water. pH was possibly over 12, maybe a bit excessive.
- 2) Extract with 150, then 100, and then 80 mL in a separatory funnel. (**330 mL of DCM**), Will use a 1 L beaker for the reaction.
- 3) Prepare an eluent for the reaction. (Read preface)
- 4) Dissolve a **1.2x molar equivalent** (to the 2C-H) of iodine in methanol, **0.156 mol or 40.2 g**. If I recall, only with difficulty did the iodine in small bead form dissolve in 220 mL methanol. A bit of undissolved iodine was on the bottom of the beaker. Used stirring and crushing with a glass stir rod to aid in dissolution. However, this is too much iodine, use a 1.05x molar amount instead. As well it could be a good idea to crush the iodine in a mortar and pestle beforehand. The methanolic solution of iodine will be very dark coloured, hard to see if there are any particles on the bottom of the beaker. Light will not pass through. But there was undissolved iodine left over and I decanted pouring into an addition funnel.
- 5) In a darkened room, cover a 1 L beaker (or a larger amber reagent bottle, or a 2L Erlenmeyer flask) with foil with the freebase 2C-H/ DCM in it. Weigh out a **1.2x molar equivalent** (to the 2C-H) of silver mesylate, **0.156 mol or 32.15 g**.
- 6) Now dissolve the silver salt in DCM, could be I used about **300 – 350 mL DCM** trying to dissolve, but some powder remained on the bottom. Maybe it would have dissolved fully with 400 mL DCM and longer stirring. Pour all of it as is into the beaker with the 2C-H. Silver methanesulfonate does not have the best solubility in DCM, and this adds to the volume of solvent used. It is however soluble in methanol...
- 7) From an addition funnel, drip in the iodine at a medium rate. Done in the dark with strong stirring in the beaker. Very quickly there is precipitation, but this is not 2C-I. It seems that 2C-I remains as a freebase dissolved in the DCM. Or is it?

What precipitates out is AgI, which is not soluble in the reaction mixture.

The reaction that occurs as outlined in the “*Selective C–H Iodination of (Hetero)arenes*” paper is that silver methanesulfonate reacts with iodine which at once forms silver iodide (AgI) and methanesulfonyl iodide ($\text{CH}_3\text{IO}_3\text{S}$). It is the methanesulfonyl iodide which reacts with the substrate arene. The one that “hands

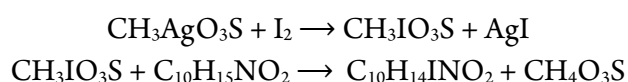
over” the Olympic torch as it were to the 2C-H, knocking off a hydrogen clumsily, like some pathetic molecular “wardrobe malfunction”.

In the vernacular, it is an electrophilic reaction whereby the I^+ latches onto the *para* position between the two electrophilic methoxy groups. Specifically what occurs is an electrophilic aromatic substitution reaction (SEAr), and an intermediate HI (hydrogen iodide) could be produced as well, but methanesulfonic acid should be formed in the end, along with the iodinated substrate.

Remember that the MSA is a liquid, miscible with the methanol.

But in turn, could it be that 2C-I mesylate is then formed? But that should be soluble in the mixture...

So working things out, the equation for the reaction is:



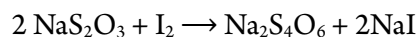
Coming back to the reaction, it is difficult to monitor any changes, but I let it spin at room temperature for 20 hours, going visually it seems as if one could get away with far less, maybe 4 hours, as there is little change. No temperature increase with addition of the iodine.

The yellowish silver iodide just spins on the bottom of the murky iodine rich mixture, and one could really monitor by colour change if one did not use an excess of iodine. The iodine is consumed with stoichiometric amounts.

7) At this point I filter off the impure Ag salts on the bottom of the beaker, wash I believe with a bit of IPA, and dry separately. The total weight of the **recovered Ag salts is 38.69 g**. If we used 0.156 mol of silver mesylate, should end up with the same molar amount of silver iodide in moles. 0.156 mol of AgI is 36.62 g, so what I collected is certainly impure and contains some silver mesylate as well. The silver mesylate was visibly whiter when in the bottom of the beaker at the beginning. (*Actually it seems I filter after adding sodium thiosulfate solution. But better to filter first and skip the next step.*)

8) Decide to take care of the excess iodine with sodium thiosulfate solution. 2 mol react with 1 mol iodine to reduce to iodide ions.

The equation for this is:



So to reduce 40 g I_2 (0.156 mol) need 0.312 mol, or 49.3 g, but decide to dissolve 25 g in 200 mL water, pour to 100 mL, but already after pouring in 50 mL or so, the mixture becomes clear, this is 6.25 g sodium thiosulfate, working this out by moles, enough to reduce 5 g I_2 . Coming to the beginning, we used 40.2 g I_2 , but a stoichiometric amount was closer to 33 g, so it could very well be that the reaction went well.

9) Decide to check the pH and it was 3 – 3.1 as I recall. The plan now is to form 2C-I acetate with addition of acetic acid (GAA). However I am sent into a panic at those numbers scurrying on my pH

probe. Only now am I working out what really went on in the reaction. It shouldn't come as a surprise, both the hydrogen iodide, and more likely the methanesulfonic acid present would lower the pH of the reaction mixture. A pH of 3 is not awfully low, so not much MSA present then?

But the methanesulfonic acid is a strong one, with a pK_A lower than that of acetic acid, so most probably it is wise to freebase at this point in order to create 2C-I acetate. As well, by raising the pH of what is now a DCM, methanol, and water mixture (with 2 layers), the 2C-I will certainly all migrate away from the MeOH + H₂O layer into the DCM layer. It could very well be that there is some 2C-I in the DCM as well. Well that is my reasoning now at least.

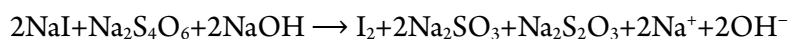
We are aiming to make 2C-I acetate because it will be soluble in the water and methanol mix, 2C-I HCl, is hardly soluble, and would pop out and float around among the junk. 2C-I formate can be made as well.

10) Of note, is that initially the volume of the mixture was 850 or 920 mL, but after 20 hours of intense mixing in a darkened bathroom, the layer is now a slimmer 650 or 700 mL (before adding thiosulfate solution).

Still going strong though...

Add aq. NaOH and raise the pH to 11.6. And both layers darken! What happens is the reverse of what adding sodium thiosulfate did. God damn it! What just happened here Frank? It seems that from Na₂S₄O₆ (sodium tetrathionate), with addition of the ⁻OH sodium thiosulfate was formed again, along with sodium sulfite and I₂.

This reaction:



Raising the pH just returned the iodine I (I and I) was looking to hide. A sort of Chernobyl-esque hide and seek with the prize of KI.

11) Now I separate the lower DCM layer from the disgusting upper aqueous layer and nuke it with dilute GAA. My actions would make any world leader proud.

More specifically, once the DCM is separated, leave it in the sep. funnel and calculate a 0.13 mol amount or 7.4 mL GAA diluted to 80 mL or so. Pour in with no remorse (but not all of it) and shake in the funnel, the pH falls to somewhere in the 4s. It is slow to separate, must be lots of product in the wee tippie of water. Decide to dilute the remaining GAA solution a bit, and extract a second time. This time the pH falls to 6.4, and I shake like no tomorrow. Alas, an emulsion forms, the layers are separating but oh so slowly, don't have the patience for this B.S anymore.

12) Salt (NaCl) – only kosher, to the rescue, pour in a heaping teaspoon, and all sorts of messed up buffoonery occurs. The products go AWOL from ALL of the layers, and both the DCM and the wee upper water layer coming into existence, is sullied and corrupted by sodium salts of god knows what orientation.

1E) Now all is lost or is it? We still have that one extract of slightly pink, light red, aqueous layer. Looks clear, probably no hope left of recovering anything, so screw it, just bring it on, bring on the concentrated HCl.

But first transfer to a 1 L beaker and heat it at 80 °C under stirring to bubble off most of residual DCM.

14) Calculate **0.12 mol HCl**. Mind you, I am still 0.12% hopeful. Of the 36% with a molarity of 11.64 mol/L makes for 10.3 mL diluted to about 100 mL. Pour in whatever amount, and stare intently.

Jah all mighty, it is a miracle! Within 2 or 3 seconds the layer goes cloudy and suddenly it solidifies. The beaker is all mushy product of what must be 2C-I HCl! I look at the pH and it is far too low, at about 0.5. There is the unpleasant scent of HCl and the speculative 2C-I is a bit pink, a sure sign of over – acidification. Well now I really did hurry. Should have left a portion out as always, and repeated the mantra, hope dies last, take a breath, the very last step, don't screw this up, but I forgot the mantra...

15) Filter into a Büchner funnel and the mother liquor in the filter flask solidifies as well, it just needed some breathing room. So I should have diluted the HCl even further, or better yet extracted from the DCM with more water, might have prevented an emulsion as well. Now I collect the filtrate and discard it, but another mistake, should have evaporated the water to collect any more possible product, it is at a low enough pH. 2C-I HCl it turns out *does* dissolve somewhat in water.

Wash what I have with water and room temperature ethyl acetate (EtOAc). Not the best choice either, would have been better to wash with only cool EtOAc to prevent product loss.

16) Fan dry the product and end up with **22.044 g** of an off-white (could call it pinkish) fine, light and fluffy powder.

If the mol. weight of 2C-H (hemi)sulfate is 230.275 g/ mol, the mol weight of 2C-I HCl is 343.588 g/mol, then I have 0.064 mol or a 49.4% yield. I will give myself a **50%** in commiseration.

The iodine in the 2C-I is very heavy, so to go from 30 g 2C-H to 30 g 2C-I you need only a 59% yield. And with Ag salt facilitated iodination, yields of 80% have been reported, so it could be that my work-up and techniques were not the best, but for a first time I will take it.

Just remember to go slow during salting.

17) Later on, fill a thiele tube with paraffin oil. Take a small sample packed down in a capillary tube, heat the thiele tube with a camping stove, aiming for 240 – 260 °C as the **melting point** of 2C-I is variously reported to be about **246 – 247 °C**. Much higher than any other substance I have tested. The thermometer I am using goes up to 250 °C as well.

At the 220 °C the paraffin really starts to smoke away, and as it heats it expands and some dribbles over one of the tubes to place in the capillary tube. It catches on fire, blow it out, but it spooks me, so I continue heating but at too fast of a pace, and notice the sample melting to disintegration only between 253 and 256 °C or so. The rate of increase was too much to give an accurate reading. But this is far above the m.p. of 2C-H HCl (138 °C) and close enough, so it should be 2C-I.

Repeated bioassays confirm that it is a totally awesome substance.

Closing Time and Photos

Turns out that it is possible to iodinate one of these freebase phenethylamines. Not necessary to go with the salt form. However DCM is not the best solvent for the experiment. If reacting say, 150 g 2C-H, would need to use 4.2 L or more of the reaction mixture.

Originally this method was written on hyperlab.info
www.hyperlab.info/inv/index.php?s=&act=ST&f=17&t=730&st=80

here is a write-up that uses ethylene glycol, and silver nitrate dissolves in it well.

As well, from the seemingly extinct www.thevespiary.org, there are a few more write-ups, along with the use of different silver salts. Search for the thread "2C-I synth with silver nitrate in MEG". Best of luck.



Set-up for the preparation of silver mesylate. Ice cooling, and an amber bottle



KI iodide oxidant test, clear solution discolours in presence of H_2O_2



Reaction in the dark. Dripping in iodine, solution not clear as some silver salts were not dissolved.



Reaction before filtering, but after addition of sodium thiosulfate, upper layer is methanol + water (from the thiosulfate sol.) lower one is the DCM layer. On the bottom are silver salts



Filtered light yellow silver species, mostly AgI



After isolating the 2C-I acetate in water, before addition of HCl



The final product drying, bit lighter in colour than in this photo